PATENT SPECIFICATION

656,405

Date of filing Complete Specification Dec. 8, 1948.

Application Date Dec. 9, 1947. *No. 32396/47.*

Complete Specification Published Aug. 22, 1951.

Index at acceptance.—Classes 1(i), F3b1, F3b2(a: x); and 2(iii), C3a13a3, C3a14a1(b: c), C3a14a8d.

PROVISIONAL SPECIFICATION

Manufacture of Methyl-Isobutyl Ketone

We, THE DISTILLERS COMPANY LIMITED, a British Company, of 12, Torphichen Street, Edinburgh, Scotland, FREDERICK

vapours are passed at 200° C. over a catalyst made up of thorium oxide, copper and nickel methyl isobutyl carbinol

SPECIFICATION NO. 656405

INVENTORS:- FREDERICK WILLIAM MAJOR and FRANCIS EDWARD SALT

By a direction given under Section 17(1) of the Patents Act 1949 this application proceeded in the name of The Distillers Company Limited, a British Company, of 12, Torphichen Street, Edinburgh 3, Scotland.

THE PATENT OFFICE,
5th November, 1951

DS 98617/1(13)/3091 160 10/51 R

with smaller quantity of methyl isobutyl ketone by the partial hydrogenation of the mesityl oxide in contact with the nickel.

In British Patent Specification No. 400,384 it is mentioned that methyl-isobutyl ketone may be produced by the condensation of acetone through mesityl oxide as an intermediary when a mixture of ketones, for instance acetone, and primary aliphatic alcohols in conjunction with diluting gases, such as hydrogen or nitrogen is passed at temperatures ranging from 150° to 400° C. over a catalyst consisting of aluminium oxide with or without magnesia or other alkaline earth oxides, supplemented by a relatively small amount of copper and/or silver or an oxide or oxides thereof with or without the addition of molybdenum trioxide. The formation of methyl isobutyl ketone, however, is a secondary reaction and subordinated to the production of still higher ketones. As chief product condensation product of the ketone with the aliphatic alcohol are obtained, besides considerable amounts of polysubstituted high boiling point ketones.

When according to British Patent Specification No. 326,812 isopropyl alcohol

processes wherein methyl isobutyl ketone is formed in small quantities as a secondary by-product, nor the circuitous method wherein mesityl oxide is formed from acetone as an intermediate, which subsequently is reduced to methyl-isobutyl ketone, offers a simple and cheap manner of manufacturing the latter compound on a commercial scale.

It is an object of the present invention to provide a process by which methyl-isobutyl ketone results as the main product. It is another object to produce this substance by a simple and easy method; it is a further object to carry through the manufacture of said product in a single stage process and with high yields, so that the process can be carried out with advantage on a commercial scale.

According to the present invention methyl-isobutyl ketone is manufactured by passing acetone vapours in conjunction with hydrogen over a catalyst which comprises essentially a mixture of metallic copper and magnesium oxide, at a temperature ranging from 180° to 300° C. preferably from 200° to 250° C. The catalyst is preferably prepared from a mixture of copper oxide and magnesite.

The catalyst is advantageously pre-

PATENT SPECIFICATION

656,405

Date of filing Complete Specification Dec. 8, 1948.

Application Date Dec. 9, 1947. *No. 32396/47.*

Complete Specification Published Aug. 22, 1951.

Index at acceptance.—Classes **1(i)**, F3b1, F3b2(a: x); **and 2(iii)**, C3a13a3, C3a14a1(b: c), C3a14a8d.

PROVISIONAL SPECIFICATION

Manufacture of Methyl-Isobutyl Ketone

We, THE DISTILLERS COMPANY LIMITED, a British Company, of 12, Torphichen Street, Edinburgh, Scotland, FREDERICK WILLIAM MAJOR and FRANCIS EDWARD SALT, both British Subjects and both of the Company's Research and Development Department, Great Burgh, Epsom, Surrey, do hereby declare the nature of this invention to be as follows:—

This invention relates to the manufacture of methyl-isobutyl ketone from acetone.

Mailhe and de Godon have described in Bull. Soc. Chim. 1917 (4) 21 61, that when acetone and hydrogen are passed over metallic nickel at a temperature of 300° C. a small quantity of mesityl oxide is produced and, admixed therewith, a still smaller quantity of methyl-isobutyl ketone by the partial hydrogenation of the mesityl oxide in contact with the nickel.

In British Patent Specification No. 400,384 it is mentioned that methyl-isobutyl ketone may be produced by the condensation of acetone through mesityl oxide as an intermediary when a mixture of ketones, for instance acetone, and primary aliphatic alcohols in conjunction with diluting gases, such as hydrogen or nitrogen is passed at temperatures ranging from 150° to 400° C. over a catalyst consisting of aluminium oxide with or without magnesia or other alkaline earth oxides, supplemented by a relatively small amount of copper and/or silver or an oxide or oxides thereof with or without the addition of molybdenum trioxide. The formation of methyl isobutyl ketone, however, is a secondary reaction and subordinated to the production of still higher ketones. As chief product condensation product of the ketone with the aliphatic alcohol are obtained, besides considerable amounts of polysubstituted high boiling point ketones.

When according to British Patent Specification No. 326,812 isopropyl alcohol vapours are passed at 200° C. over a catalyst made up of thorium oxide, copper and nickel, methyl isobutyl carbinol results as the main product and in excellent yield. Methyl isobutyl ketone is only produced as a by-product.

It is furthermore known through British Patent Specification No. 362,204 that methyl isobutyl ketone can be produced by first converting acetone into diacetone alcohol which is subsequently converted into mesityl oxide. Vapours of this latter substance are then passed with excess of hydrogen over a catalyst composed of reduced nickel or copper, whereby it is hydrogenated and converted into methyl isobutyl ketone.

From this it is clear that neither the processes wherein methyl isobutyl ketone is formed in small quantities as a secondary by-product, nor the circuitous method wherein mesityl oxide is formed from acetone as an intermediate, which subsequently is reduced to methyl-isobutyl ketone, offers a simple and cheap manner of manufacturing the latter compound on a commercial scale.

It is an object of the present invention to provide a process by which methyl-isobutyl ketone results as the main product. It is another object to produce this substance by a simple and easy method; it is a further object to carry through the manufacture of said product in a single stage process and with high yields, so that the process can be carried out with advantage on a commercial scale.

According to the present invention methyl-isobutyl ketone is manufactured by passing acetone vapours in conjunction with hydrogen over a catalyst which comprises essentially a mixture of metallic copper and magnesium oxide, at a temperature ranging from 180° to 300° C. preferably from 200° to 250° C. The catalyst is preferably prepared from a mixture of copper oxide and magnesite.

The catalyst is advantageously pre-

pared by incorporating copper formate into magnesite and roasting said mixture at a temperature of about 400° C. for a period of several hours and thereafter subjecting the product thus obtained to a reduction treatment by means of hydrogen or gases or substances generating hydrogen under reaction conditions. Reduction may be carried out preferably in two stages in which hydrogen is first passed for some time over the mixture at a temperature of 250° C. which temperature is then raised to about 400° C., at which temperature it is maintained again for some time. The first stage of the reduction at 250° C. is maintained with advantage for about 4 hours, whilst during the second stage the temperature is kept at 400° for about 3 hours.

When a mixture of acetone vapours with hydrogen in a proportion of about 2 molecules of acetone and one molecule of hydrogen is passed over the catalyst prepared as described above, approximately 70 to 75% of the acetone reacts by condensation and hydrogenation in a single pass. 50 to 60% of the acetone converted forms methyl isobutyl ketone, and 20% is converted into di-isobutyl ketone. Part of the remaining 20 to 30% is converted into higher condensation products. Most of the acetone which does not react may be recovered as such, whilst a very small proportion is converted during the reaction into gaseous products such as hydrocarbons.

An increase of the reaction temperature beyond the temperature limit of 300° C., is unfavourable for the production of methyl isobutyl ketone, since it results in increasing the production of high boiling condensation products.

The process according to the invention may be carried out at atmospheric, sub-atmospheric or super-atmospheric pressure.

When in the course of the reaction the catalyst loses its activity it can be revivified by subjecting it to a heat treatment in the presence of oxygen-containing gas and a subsequent reduction by means of hydrogen. The oxidation is preferably carried out like the initial preparation of the catalyst, at about 400° C. and the reduction treatment preferably in two stages at 250° and 400° C. respectively as before described, whereby the catalyst regains substantially its previous activity.

A number of comparative tests were carried out with a catalyst which was prepared from a mixture of copper oxide and magnesite or magnesium oxide respectively on the one hand and copper on aluminium oxide on the other hand. These tests have shown that a considerable proportion of acetone was converted into hydrocarbons and into condensation products of higher molecular weight when copper on aluminium oxide is used, compared with the catalyst according to the process of the present invention. In tests performed with a separate nickel catalyst a total conversion of the acetone amounted to only 45 to 50% of that obtained when the catalyst was prepared from a mixture of copper oxide and magnesite.

The following example illustrates the manner in which the invention may be carried out:—

EXAMPLE.

A mixture of magnesite and copper oxide in such proportions that the ultimate product contained about 3 to 4% of the copper compound calculated as metallic copper, was treated for 2 hours at 400° C. whilst air was conducted over it and then subjected to a reduction treatment which consisted in passing hydrogen over it at a temperature of 250° C. This temperature was raised after four hours to about 400° C. and there maintained for approximately three hours.

300 cc. of this catalyst were placed in a mild steel tube and maintained at a temperature of 250° C. whilst a mixture consisting of acetone vapours and hydrogen in the molecular proportion of 2:1, was passed through the tube the acetone rate being 0.065 litre of liquid and the hydrogen rate 11.4 litres (NTP) per hour. During the period from 2 to 7 hours after the commencement 70% of the acetone introduced into the contact vessel reacted. The gaseous reaction mixture was cooled and condensed and the unreacted acetone distilled off. The acetone-free reaction product consisted of:—

Methyl isobutyl ketone -	44% w/w
Isopropyl alcohol - - -	4% w/w
Di-isobutyl ketone - - -	17% w/w
Methyl isobutyl carbinol -	1% w/w
Higher products - - - -	15% w/w
Water - - - - - - - -	19% w/w

In this instance, the production of methyl isobutyl ketone amounted to 56 grams per hour per litre of catalyst.

The catalyst remained active for a considerable time.

When a similar mixture of acetone vapours with hydrogen was passed over a mixture of elemental copper and aluminium oxide instead of magnesite as catalyst under the same conditions as stated above, a slightly higher conversion of acetone was obtained, but with greatly diminished production of methyl isobutyl ketone. In addition, considerable amounts of hydrocarbons were produced with much larger quantities of methyl isobutyl carbinol. The composition of the

product calculated on an acetone-free basis was as follows:—

Methyl isobutyl ketone	11%
Di-isobutyl ketone	12%
Methyl isobutyl carbinol	13%
Higher condensation products	19%
Liquid hydrocarbons	7%
Condensed gaseous products	7%
Water	31%

The copper plus aluminium oxide catalyst gave a rate of production of methyl isobutyl ketone of only 12 grams per hour per litre of catalyst, compared with the value of the 56 grams quoted above for the catalyst prepared from copper oxide and magnesium according to the present invention.

Methyl isobutyl ketone may be used as a solvent or as an intermediate. The di-isobutyl ketone may also be used for the same purposes.

Dated this 5th day of December, 1947.

N. F. BAKER,
Agent for the Applicants.

COMPLETE SPECIFICATION

Manufacture of Methyl-Isobutyl Ketone

We, THE DISTILLERS COMPANY LIMITED, a British Company, of 12, Torphichen Street, Edinburgh, Scotland, FREDERICK WILLIAM MAJOR and FRANCIS EDWARD SALT, both British Subjects and both of the Company's Research and Development Department, Great Burgh, Epsom, Surrey, do hereby declare the nature of this invention and in what manner the same is to be performed, to be particularly described and ascertained in and by the following statement:—

This invention relates to the manufacture of methyl-isobutyl ketone from acetone.

Mailhe and de Godon have described, in Bull. Soc. Chim. 1917 (4) 21 61, that when acetone and hydrogen are passed over metallic nickel at a temperature of 300° C. a small quantity of mesityl oxide is produced and, admixed therewith, a still smaller quantity of methyl-isobutyl ketone by the partial hydrogenation of the mesityl oxide in contact with the nickel.

In British Patent Specification No. 400,384 it is mentioned that methyl-isobutyl ketone may be produced by the condensation of acetone through mesityl oxide as an intermediary when a mixture of ketones, for instance, acetone, and primary aliphatic alcohols in conjunction with diluting gases, such as hydrogen or nitrogen, is passed at temperatures ranging from 150° to 400° over a catalyst consisting of aluminium oxide with or without magnesia or other alkaline earth oxides, supplemented by a relatively small amount of copper and/or silver or an oxide or oxides thereof with or without the addition of molybdenum trioxide. The formation of methyl isobutyl ketone, however, is a secondary reaction and subordinated to the production of still higher ketones. As chief product condensation products of the ketone with the aliphatic alcohol are obtained, besides considerable amounts of polysubstituted high boiling point ketones.

When according to British Patent Specification No. 326,812 isopropyl alcohol vapours are passed at 200° C. over a catalyst made up of thorium oxide, copper and nickel, methyl isobutyl carbinol results as the main product and in excellent yield. Methyl isobutyl ketone is only produced as a by-product.

It is furthermore known through British Patent Specification No. 362,204 that methyl isobutyl ketone can be produced by first converting acetone into diacetone alcohol which is subsequently converted into mesityl oxide. Vapours of this latter substance are then passed with excess of hydrogen over a catalyst composed of reduced nickel or copper, whereby it is hydrogenated and converted into methyl isobutyl ketone.

From this it is clear that neither the processes wherein methyl isobutyl ketone is formed in small quantities as a secondary by-product, nor the circuitous method wherein mesityl oxide is formed from acetone as an intermediate, which subsequently is reduced to methyl-isobutyl ketone, offers a simple and cheap manner of manufacturing the latter compound on a commercial scale.

It is an object of the present invention to provide a process by which methyl-isobutyl ketone results as the main product. It is another object to produce this substance by a simple and easy method; it is a further object to carry through the manufacture of said product in a single stage process and with high yields, so that the process can be carried out with advantage on a commercial scale.

According to the present invention methyl isobutyl ketone is manufactured by contacting acetone vapours and hydrogen at a temperature between 180° and 300° C. with a contact material which comprises initially mixture of a copper compound which is reducible to metallic copper by the mixture of said reactants at the reaction temperature, with magnesium oxide and/or magnesium hydroxide. The mixture of acetone and

hydrogen may be conducted over the solid contact material or the latter may be dispersed in a liquid inert medium which is relatively non-volatile under the reaction conditions. Suitable inert media are for instance high boiling hydrocarbons like kerosene or paraffin oil, vegetable oils such as cotton seed oil, liquid silicones and high boiling polyglycol ethers.

Copper compounds reducible by the mixture of the reactants are for instance cupric oxide, cuprous oxide and the hydroxides of copper. The copper compound preferably used in the initial contact material is cupric hydroxide and the initial contact material is most conveniently prepared by mixing an aqueous solution of a water-soluble copper salt such as the formate, nitrate or acetate with an excess of magnesium oxide or hydroxide. The magnesium oxide may be prepared from precipitated hydroxide or it may be calcined magnesium carbonate, either in the form of fine powder or of granules, which latter, when brought into contact with the copper solution, are covered and impregnated by the copper compound formed. The initial mixture may also be prepared by thoroughly mixing the hydroxide and/or oxide of magnesium with a suitable copper compound such as the oxide or hydroxide of copper in the dry state.

When the mixture obtained by the mixing of said compounds if necessary after having been dried is exposed without any further treatment to the action of a mixture of acetone vapours and hydrogen at a temperature between 180° and 300° C. according to the process of this invention the conversion of the acetone and the formation of methyl isobutyl ketone very soon begins. It is preferred, however, to give the contact material a preliminary treatment by heating the above mixture or the mixture obtained by the interaction of the copper salt and an excess of the magnesium compound and after removal of the water soluble magnesium salt in a current of hydrogen before the contact material is brought into contact with the acetone-hydrogen mixture. The heat treatment in the current of hydrogen alone may be carried out at a temperature of between 250° and 500° C. preferably between 350° and 450° C. In some cases it was found to be advantageous to carry out the heat treatment in hydrogen in two stages, namely, firstly at a comparatively low temperature of about 250° to 300° C. and secondly at a temperature of about 400° to 500° C. It was found, furthermore, that advantages, for instance regarding the life or activity of the contact material, may be obtained when the mixture of copper compound and magnesium compound before being heated in a current of hydrogen, as set out before, is calcined by heating the mixture to a temperature between 300° and 500° C. prior to the reduction treatment with hydrogen.

The various treatments with hydrogen at the different high temperature ranges in the preparation of the catalyst are preferably carried out for several hours varying from two to five hours.

The composition of the catalyst has not been established with certainty. It is most likely that most of the copper is present during the reaction in the form of metal and the magnesium in the form of its oxide. It has been found, however, that even after prolonged runs of the actual process there is still some magnesium hydroxide contained in the contact material when magnesium hydroxide was present in the initial mixture.

The proportion of copper in the contact material to the amount of magnesium oxide or magnesium hydroxide contained therein may vary within wide limits. It was found that a proportion of 2 to 4% by weight of elemental copper in the contact material gave very good results.

The preferred temperature range for the reaction of the acetone with hydrogen in contact with the catalytic material is 200° to 250° C. and the molecular ratio of acetone vapours and hydrogen in the mixture which is passed over the contact material is with advantage about 2 molecules of acetone to about one molecule of hydrogen. When such a mixture is passed over the contact material prepared as described above, approximately 70 to 75% of the acetone reacts by condensation and hydrogenation in a single pass. 50 to 60% of the acetone converted forms the methyl isobutyl ketone, and 20% is converted into di-isobutyl ketone. Part of the remaining 20 to 30% is converted into higher condensation products. Most of the acetone which does not react may be recoverd as such whilst a very small proportion is converted during the reaction into gaseous products such as hydrocarbons.

An increase of the reaction temperature beyond the temperature limit of 300° C. is unfavourable for the production of methyl isobutyl ketone, since it results in increasing the production of high boiling condensation products.

The process according to the invention may be carried out at atmospheric, subatmospheric or super atmospheric pressure.

An appropriately prepared catalyst has been used in the process for more than 100

hours. When its activity has declined to such an extent that the reaction becomes uneconomical the catalyst can be revivified by subjecting it to a heat treatment in the presence of molecular-oxygen containing gases such as air and a subsequent reduction by means of hydrogen. The first of these stages, namely the heat treatment in the presence of oxygen is carried out between about 250° C. and 500° C. and preferably between 350 and 450° C. and the reduction treatment preferably in two stages at about 250° C. and between 400° to 500° C. respectively, as before described. The contact material hereby regains substantially its previous activity.

The following examples illustrate the manner in which the invention may be carried out.

EXAMPLE 1.

A solution of 22.9 grams of cupric nitrate tri-hydrate in 700 cc. of water was added to a stirred suspension of 175 grams light magnesium oxide in 3.5 litres of cold water. The resulting pale blue precipitate was filtered, washed with water and dried below 100° C. The cake was broken up and sieved to 4 to 10 mesh granules.

The mixture of copper hydroxide and magnesium hydroxide thus obtained contained 2.27% of copper, calculated as copper metal. 300 cc. of the dried mixture was placed in a mild steel tube. It was then heated and maintained at a temperature of 300° C. whilst a mixture consisting of acetone vapours and hydrogen in the molecular proportion of 2:1 was passed through the tube, the acetone rate being 80 cc. of liquid and the hydrogen rate 11.4 litres (NTP) per hour. The product collected from the second to seventh hour of the run was distilled and yielded 226 grams of unchanged acetone. The remainder was found to have the following composition, in percentages by weight:—

Methyl isobutyl ketone - - -	23.5%
Mesityl oxide - - - - - -	19.8%
Isopropyl alcohol - - - -	12.1%
Water - - - - - - - -	20.6%
Residue - - - - - - - -	24.0%

The percentage of acetone converted was 28.7%.

EXAMPLE 2.

A mixture of copper hydroxide and magnesium hydroxide prepared as described in Example 1 and containing 2.62% by weight of copper, calculated as copper metal, was treated in a stream of 0.75 mole/hour of hydrogen for 4 hours at 250° C., followed by 3 hours at 400° C. Over 300 cc. of this contact material was conducted a mixture of acetone vapour and hydrogen at a temperature of 250° C. the feed rate of the two components being 1 mole/hour of acetone and 0.5 mole/hour of hydrogen. The product collected from the second to the seventh hour amounted to 286 grams which separated into two phases. By fractionation 84 grams of unchanged acetone were obtained; the remainder had the following composition:—

Methyl isobutyl ketone - - -	38.6%
Di-isobutyl ketone - - - -	22.2%
Isopropyl alcohol - - - -	3.1%
Water - - - - - - - -	20.7%
Residue - - - - - - - -	15.4%

The percentage of acetone converted was 72%.

EXAMPLE 3.

A mixture prepared by mixing calcined magnesite with copper formate dissolved in sufficient water to wet the whole of the material, was, after drying, heated in a current of air for 1 hour at 400° C. The material then contained 2.19% by weight of copper calculated as metal. 300 cc. of it was passed into a mild steel tube and heated to a temperature of 250° C. A mixture of acetone vapour and hydrogen in the same proportion and at the same rate as described in Example 2 was passed over this contact material at 250° C. 296 grams of condensate were collected between the second and seventh hour of the run. This contained 158 grams of unchanged acetone and the remaining mixture had the following composition:—

Methyl isobutyl ketone - - -	39.5%
Di-isobutyl ketone - - - -	15.0%
Isopropyl alcohol - - - -	15.5%
Methyl isobutyl carbinol - -	1.8%
Water - - - - - - - -	17.0%
Residue - - - - - - - -	11.2%

The percentage of acetone converted was 45.4.

EXAMPLE 4.

300 cc. of a mixture prepared as described in Example 3 and heated to 400° C. for 1 hour were packed into the reaction tube and treated at 500° C. for two hours in a stream of 0.75 mole/hour of hydrogen. After cooling and reheating to 250° C. a mixture of acetone vapour and hydrogen in the described ratio of Example 2 was passed at 250° C. over the catalyst for seven hours. 328 grams of a product were collected between the second and seventh hour which yielded 150 grams of unchanged acetone. The remainder had the following composition:—

Methyl isobutyl ketone - - -	45.4%
Di-isobutyl ketone - - - -	11.5%
Methyl isobutyl carbinol - -	1.8%

Isopropyl alcohol - - - -	7.2%
Water - - - - - - - - -	19.9%
Residue - - - - - - - -	14.2%

The percentage of acetone converted was 52.5.

EXAMPLE 5.

The same catalyst as used in Example 4 was used for further conversions during 79 hours, when its activity showed a considerable slowing down. It was regenerated by passing air over the bed for 5 hours at 500° C. at the rate of 75 litres per hour (NTP). Subsequently, hydrogen at the rate of 0.75 moles per hour was passed for 2 hours at 500° C. thereover. The contact material was then cooled in hydrogen to 250° C. and used for a further reaction with acetone vapours and hydrogen, as described in Example 4. The product collected in the period from the second to seventh hour amounted to 263 grams, which contained 96 grams of unchanged acetone. The remainder had the following composition:—

Methyl isobutyl ketone - - -	35.4%
Di-isobutyl ketone - - - -	12.0%
Methyl isobutyl carbinol - -	2.2%
Isopropyl alcohol - - - -	7.4%
Water - - - - - - - - -	20.3%
Residue - - - - - - - - -	22.7%

The percentage of acetone converted was 62.6.

From the above examples it can be seen that production of methyl isobutyl ketone per hour per litre of catalyst may amount to as much as 55 grams.

When a mixture of acetone vapours and hydrogen similar to that used in the above examples was passed over a mixture of elemental copper and aluminium oxide under the same conditions as stated above, a slightly higher conversion of acetone was obtained but with greatly diminished production of methyl isobutyl ketone. In addition, considerable amounts of hydrocarbons and condensation products of higher molecular weight were produced with much larger quantities of methyl isobutyl carbinol. The composition of the product calculated on an acetone-free basis was as follows:—

Methyl isobutyl ketone - - -	11.0%
Di-isobutyl ketone - - - -	12.0%
Methyl isobutyl carbinol - -	13.0%
Higher condensation products	19.0%
Liquid hydrocarbons - - -	7.0%
Condensed gaseous products -	7.0%
Water - - - - - - - - -	31.0%

The catalyst containing elemental copper and aluminium oxide gave a rate of production of methyl isobutyl ketone of only 12 grams per hour per litre of catalyst compared with a value of up to 55 grams quoted above from the catalyst according to the present invention.

In tests performed with a separate-nickel-magnesia catalyst a total conversion of acetone amounted to only 45 to 50% of that obtained when the catalyst consisted initially of copper oxide in admixture with magnesium oxide and/or hydroxide.

Methyl isobutyl ketone may be used as solvent or as an intermediate. The di-isobutyl ketone may also be used for the same purpose.

In British Patent No. 410,148 there is described a process for the hydrogenation of organic compounds in the liquid phase by means of catalysts which comprise oxides of copper and chromium which also contain oxides of alkali metals and/or of alkaline earth metals, amongst which magnesium is specified. When acetone is subjected to the claimed treatment isopropyl alcohol is obtained whereas isobutyl methyl ketone yields 2-methyl-pentanol-4. In contrast, the present invention deals with the hydrogenation in the vapour phase with the aid of a catalyst which contains only copper oxide as heavy metal compound.

Having now particularly described and ascertained the nature of our said invention and in what manner the same is to be performed, we declare that what we claim is:—

1. Process for the manufacture of methyl isobutyl ketone which comprises contacting acetone vapours and hydrogen at a temperature between 180° and 300° C. with a contact material comprising initially a mixture of a copper compound, which is reducible to metallic copper by the mixture of said reactants at the reaction temperature, with magnesium oxide and/or magnesium hydroxide.

2. Process according to claim 1, wherein the copper compound is cupric hydroxide.

3. Modification of the process according to claims 1 or 2, wherein the contact material has been subjected to a preliminary reduction by heat treatment in the presence of hydrogen.

4. Process according to claim 3, wherein the reduction treatment is carried out at a temperature between 250° and 500° C.

5. Process according to claim 3 or 4, wherein the initial contact material is calcined at a temperature between 300° and 500° C. prior to the reduction treatment with hydrogen.

6. Process according to any one of the preceding claims, wherein the temperature at which the acetone and hydrogen are brought in contact with the contact material is between 200° and 250° C.

7. Process according to any of the preceding claims, wherein the acetone and

hydrogen are applied in a molecular ratio of two to one.

8. Process according to claims 3 and 4, wherein the contact material is prepared by mixing an aqueous solution of a copper salt with an excess of magnesium oxide, removing the water-soluble magnesium salts, drying the mixture containing copper hydroxide and magnesium hydroxide thus obtained and subjecting it to the reduction treatment in the presence of hydrogen.

9. Process for the preparation of methyl isobutyl ketone according to claim 3, in which the contact material is prepared by heating a mixture containing copper oxide, magnesium oxide and/or magnesium hydroxide in the presence of hydrogen to a temperature of between 250° and 500° C.

10. Process according to any of the preceding claims in which the catalyst is revivified after use by heating it in the presence of molecular oxygen containing gases at a temperature between 250° and 500° C.

11. Process for the manufacture of methyl isobutyl ketone as described in Examples 1, 2, 3, 4 or 5.

12. Methyl isobutyl ketone when produced by a process as claimed in any of claims 1—11.

Dated this 7th day of December, 1948.
N. F. BAKER.
Agent for the Applicants.

Leamington Spa: Printed for His Majesty's Stationery Office, by the Courier Press—1951.
Published at The Patent Office, 25, Southampton Buildings, London, W.C.2, from which copies, price 2s. per copy; by post 2s. 1d., may be obtained.